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Research Directed Toward Improved Echelles for the Ultraviolet

by

HYPERFINE INC.

149 N. Main Street Fairport, New York 14450

Contract NAS5-22859

FINAL REPORT

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Prepared for

Goddard Space Flight Center

Greenbelt, Maryland



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Abstract

This research was undertaken to demonstrate that improved efficiencies for low frequency gratings are obtainable with the careful application of present technology. The motivation for the study was the desire to be assured that the grating-efficiency design goals for potential Space Telescope (ST) spectrographs can be achieved. For example, one spectrograph design studied during phase B required for effective operation an echelle and predisperser grating both with a minimum efficiency of 25% at Lyman Alpha. As Hyperfine had ruled a predisperser grating for the IUE spectrograph (1° blaze, 369 gr/mm) that had an efficiency of 57% in first order at Lyman Alpha, and as the ST Phase B studies indicated the need for high efficiency gratings, the ST Project Office funded this study to develop the technology needed to produce echelles of as high efficiency as possible. The contract work was organized to compare gratings made with changes in the three spe fic parameters: the ruling tool profile, the coating material, and the lubricants used during the ruling process. A series of coatings and test gratings were fabricated and were examined for surface smoothness with a Nomarski Differential Interference Microscope and an electron microscope. Photomicrographs were obtained to show the difference in smoothness of the various coatings and rulings. Efficiency measurements were made for those test rulings that showed good groove characteristics: smoothness, proper ruling depth, and absence of defects (e.g., streaks, feathered edges and rough sides). The intuitive feeling that higher grating efficiency should be correlated with the degree of smoothness of both the coating and the grating groove is supported by the results.

I. Introduction

Through extensive discussions with Goddard Space Flight Center personnel, it was agreed that the research would be done in a series of low frequency test rulings on aluminum, aluminum-silicon alloy, and gold coatings. The coatings for ruling and the rulings actually made are listed in Appendix A. This list shows the parameters that were varied — ruling metal, deposition method, diamond tool shape, ruling lubricant, groove frequency together with coating thickness. Appendix A characterizes most of the micrographs used in the figures of this report.

The test echelles were required to have a ruled width of 30mm and a groove length of 100mm to permit replication for electron microscopy and for testing efficiency.

A groove frequency of 300 gr/mm was selected for initial work because overcoming any difficulties at this frequency was expected to guide Hyperfine in ruling 100 gr/mm echelles. The final required step in the program was to be a more restricted study of 100 gr/mm echelles.

At Hyperfine the practical goal of the present contract was to demonstrate efficiencies greater than 50% at wavelengths somewhat less than 0.2µm.

II. Grating Technology

To produce gratings of acceptable quality for vacuum ultraviolet (VUV) spectrography, the following crafts contribute significantly: figuring the master grating blank, depositing the coating on the master blank, sharpening the diamond burnishing tool, diagnostically examining test rulings, ruling the grating, replicating the grating, and testing the product replica for optical efficiency.

Blank figuring is an art with long tradition¹. Vacuum deposition techniques and equipment for making thin coatings are well described by Holland². Procedures for making coatings thicker than 1 um are still being developed. Diamond tools used for ruling gratings are prepared by specialists; Hyperfine tools were made by J. Robert Moore Co.³ Stroke⁴ has described grating technology including engine descriptions, grating and echelle theory, ruling procedure, test ruling examination by interference and by electron microscopy, optical efficiency measurement and replication of product gratings. The electron microscopy process used at Hyperfine was reported by Griffin⁵ to the Electron Microscopy Society, but the process has not been published in any journal.

An echelle grating (deep grooves, usually low frequency) can, when used in conjunction with a predisperser (shallow grooves, also low frequency) with its ruling orthagonal (crossed) to those of the echelle produce spectral orders positioned one above the other. Such an optical system has been chosen both for the two spectrographs of the IUE (International Ultraviolet Explorer⁶) and for the High Resolution and Faint Object Spectrographs of the ST (Space Telescope⁷).

Fastie and Mount⁸ have analyzed echelle grating theory and reported the state of the art as of 1976 both for UV echelles and for their predispersers. They reported efficiencies of 44% near 0.2µm for one echelle (101.95 gr/mm) and of 50% for another (63.2 gr/mm). These gratings were ruled in coatings that were probably as thick as 8µm and 12µm respectively. The microscopic appearance of these two gratings indicates that the masters were ruled in aluminum.

Harrison⁹ et al has reported on the rulability problems of Au and of Al for large gratings and echelles. Their report favors Al over Au for groove frequencies equal to or less than 300 gr/mm where the lesser cumulative groove length, despite the abrasiveness of Al, would not wear away the diamond shape significantly. Based in part on their report, Hyperfine recognized that the problems with ruling gold coatings included: (1) "light-scattering crystal structure where ruled" for some gold coatings, (2) larger loading mass required on

a diamond when ruling gold than when ruling aluminum, (3) less predictable adherence of gold to substrate. Notwithstanding these problems, Hyperfine hypothesized that higher optical efficiency could be achieved for UV echelles ruled in thick gold coatings. To accomplish this, plans were made to be able to use two different diamond radii (Fig. 6) and different ruling lubricants.

III. Coatings

IIIA. General

Coatings are normally deposited on the master blank using either resistance heating or electron bombardment heating (electron-gun). A principal advantage of the electron gun is that coating thickness is less limited by the amount of metal that can be held in the evaporant crucible. Resistance fired thick coatings use heavy current through the large charge of molten metal, use indirect heating or use a turret of filaments fired sequentially in the same pump down. Layered resistance fired coatings deposited in a succession of pump downs have delaminated during ruling. Other methods such as sputtering, induction heating, or laser heating could conceivably be used.

The chemical, structural, and defect nature of thick metal coatings has not been studied here. Hyperfine sought to obtain thick gold coatings (as well as thick aluminum coatings) suitable for ruling. The big problem for thick aluminum is surface roughness that persists to some extent after ruling; for gold it is adherence. Other problems for various metals include low malleability, diamond tool wear, and low specular reflectance after ruling, thus gold and aluminum are the most suitable master coatings known at this time. All alloyed with Si may prove better than pure Al.

IIIB. Adherence

Standard practice to improve coating adherence to glass substrates has been to deposit about 30nm of chromium metal prior to depositing aluminum¹⁰ or gold¹¹. Chromium was used as a base layer for all of the thick coatings reported in Appendix A even for the run in which an aluminum base layer was used under gold.

There is a military specification¹² for testing coating adherence using cellulose tape. The tape test Hyperfine applies to a coating prior to grating ruling is especially vigorous (yanked vs. pulled) and is repeated in a crossed direction. Vendors would not guarantee adherence of thick gold coatings. Table I summarizes quotes obtained for thick aluminum and thick gold coatings.

Table I. Prices per run quoted for thick AI and Au coatings, adherence not guaranteed. Thicknesses are in µm.

| Vendor | Al | | Au |
|--------|-------|----------|--------------|
| | Thick | Price | Thick Price |
| | | | |
| 1. | 10 | \$ 800. | 2.5 \$250.* |
| 2. | 5 | 2,000. | Declined |
| 3. | 10 | 1,500.** | - |
| 4. | | - | 10 \$500.*** |

Because no vendor would guarantee coating adhesion or rulability and because the material cost per run was a significant percentage of the entire contract price, Hyperfine found it necessary to prepare all of the thick coatings used in the latter half of this contract.

^{*}Lost all interest in making thick coatings after succesive adherence failures of 2.5µm to 3.0µm gold.

^{**}Sputtered alloy.

^{***}Quote for material cost only; provided as promotional effort by supplier of sputtering equipment.

Adherence results based on Hyperfine's experience are summarized by the following statements:

- Aluminum adherence failures are rare.
- Experience has shown that adherence failures eventually occur after some number of replications from the master.
- 3. An Alloyed aluminum-silicon 10µm coating (recommended by John Mangus of NASA) had no adherence problem.
- Gold adherence diminished as coating thickness increased; internal stresses are suspected.
- 5. Coating separation of gold usually occurs at the chromium-gold interface
- 6. The likelihood that 10µm Au coatings will endure the tape test has increased to an estimated 50%.
- 7. All of the rulings made for this contract were successfully replicated for electron microscope examination and for efficiency testing.

IIIC. Equipment

Fig. 7 through Fig. 9 are photographs of the coater (shown open), the turret filament system, and the planetary substrate holders (an echelle blank is at the top rear). The ability to rotate the master blank both around the system axis and the grating blank axis during deposition was believed escential in order to obtain good coating uniformity in accord with the principles described by Strong¹³ while inhibiting preferential dendritic growth during deposition. The turret filament system was used for the chromium base film followed by a succession of evaporant depositions. The evaporant was premelted on the filament while a shutter covered the whole assembly. This protected the blanks from any violent eruptions that occur during the initial melting phase. Once the melt had stabilized visually, the shutter was opened and evaporation begun, until the correct thickness had been achieved. The coating thickness was monitored with a commercial system that correlates the frequency change of a quartz crystal (circular unit, lower center of Fig. 9) due to the added mass deposited; this system had been cross checked to at least 2.5% for thinner films using an interference microscope.

IIID. Coating Smoothness

Electron micrographs of all of the coatings ruled in this contract are presented in Fig. 1 or Fig. 2. Fig. 1 provides a comparison of various thicknesses of AI coating deposited from resistance heated filaments or deposited using an electron-gun. Fig. 1d shows the surface of the thick AI-Si alloy coating prepared by Hyperfine. The composition of the alloy before evaporation was 98% AI and 2% Si. The final composition of the coating is not known. Fig. 2 contrasts different thicknesses of Au and AI deposited, using resistance heated filaments.

Remarkable surface differences are apparent.

- Surface roughness increases with coating thickness.
- 2. Al is rougher than the Al-Si alloy.
- 3. Both Al and the Al-Si alloy are much less smooth than Au of comparable thickness.
- 4. The one run of e-gun deposited AI was rougher than resistance fired AI. The e-gun was not used for other runs.
- 5. Diffuse reflection by Al appears visually to increase as a stronger function of thickness than for Au.

As noted in 4. above, the e-gun deposited AI coating was rougher than the resistance fired coating. However, contrary to our expectations, the grooves in the e-gun AI were smoother than those in the resistance fired AI.

IV. Rulability

IVA. General

The principal process in grating technology is ruling smooth, sharp, accurate, consistent grooves. Rulability is, granting adherence, the most significant property of the coating in which an echelle is to be ruled.

There are some internal metallurgical characteristics that make certain metals unsuitable for ruling purposes. In previous trials, palladium has seemed extremely abrasive, inconel has seemed unmalleable, and indium has seemed to undergo some changes of phase when ruled. Even gold in some instances has had inhomogeneity grains that were being dislodged and dragged by the diamond, producing ruling streaks. Aluminum and gold (when free of inhomogeneities) are excellent metal coatings in which to rule deep grooves. Hyperfine compared the rulability of these metals in this contract.

The physical process of ruling a groove is complex because material is displaced and partly raised, not removed, while the tool is dragged through the metal layer to burnish its surface. The tool is a diamond cemented to a cylindrical rod clamped in a hinged holder that can be lifted away from the grating while the tool is being returned for its next groove. Mass is added to the tool assembly to cause the tool to *float* deeper in the metal, a static condition. The ruling of each succeeding groove generates a wave (a kinetic effect) that causes some change of the optical face of the groove previously burnished. By orienting the tool optimally, it is possible to minimize this groove-to-groove interaction. Internal characteristics of the metal when stressed well beyond its elastic limit are believed to influence the extent of the displacing wave. This remote interaction of the tool on the metal could be thought of as the macroscopic aspect of rulability. Having minimized the magnitude of this ruling problem by rotating the tool on its axis, the remaining change can be compensated during set-up by changing the tilt angle of the tool.

The development of a "feather edge" or burr along the ridge between adjacent grooves could be thought of as the microscopic aspect of rulability. This burr complicates replication take-apart. Odd-generation replication product gratings invert the grooves and bury the burr to diminish its optical significance. Overloaded tools produce exaggerated "feather edges."

Another aspect of rulability is the change of surface smoothness before and after ruling. This depends at least on the initial smoothness of the unruled surface and on the malleability of the film material. The residual roughness appearing in each groove often near the top and on the unblazed face varies randomly across the grating.

The wear of the diamond tool by the metal being burnished has already been mentioned and should be classified as a rulability limitation because the last grooves can differ significantly from the first grooves. This is a systematic defect that is evidenced by progressive grating efficiency reductions. One of the expected advantages of gold over aluminum was the reported low abrasiveness of gold.

IVB. Observation of Rulability-Microscopy

There are contrasting methods of studying grating rulability problems: direct examination of the grating grooves using microscopes and indirect examination using the spectrographic performance as an indicator of the integrated average of the ruling errors. This second method is more difficult and less diagnostic, though more significant.

Individual grooves of low frequency gratings and echelles can be studied using optical interference microscopy. Hyperfine uses a Leitz Interference Microscope mainly for assaying test rulings during set-up. It can clearly show the difference between the terminal groove and the rest of the grating grooves (macroscopic problem). A Zeiss Nomarski Differential Interference Microscope is also used during set-up to assess the smoothness of the burnishing and of the unruled coating. Optical microscopy is not nearly as sensitive as electron microscopy to surface roughness and this report presents electron microscope pictures exclusively.

Aluminum oxide replicas (Griffin⁵) were used for electron microscopy of 300 gr/mm grooves and surface texture of the 2.5µm and 3µm thick coatings; conventional collodion replicas were used for 100 gr/mm grooves and for the 10µm thick coatings, because these deeper grooves and thicker coatings were too rough for non-plastic replica films.

Fig. 10 is a photograph of the inside of the coater used at Hyperfine for forming and shadowing electron micrograph replicas. Fig. 11 is a photograph of the electron microscope used for the work reported here. Fig. 12 is the projection system used to measure groove profile details.

IVC. Ruling Lubricant and Tool Shape

A puddle of fluid ordinarily is flowed across the grating blank to facilitate the burnishing process. Two lubricants were used in this work: Dow Corning Silicone 704 and Cindol 3401.

Test Echelle number 3B (300 gr/mm in 2.5µm gold) used no lubricant. Attempts to rule 100 gr/mm test echelles in gold without a liquid were unsuccessful.

The first experimental task of this contract compared the smoothness of grooves in 3µm Al when each of three variables had two options: (1) the coating was resistance fired vs. e-gun, (2) the diamond tool had an 18mm radius vs. 6mm, (3) the lubricant was Silicone 704 vs. Cindol 3401.

Figs. 3a through 3h are electron micrographs of the eight combinations of these options. These figures show:

- The 18mm radius diamond burnished a little more smoothly than the 6mm radius diamond. (the 18mm radius was used thereafter).
- Silicone appears to be as good a lubricant as Cindol. (The difference does not appear significant. Silicone was used for most of the other test echelles.)
- 3. The grooves in e-gun AI seem more fully burnished than those in the resistance AI.

All subsequent test echelles were for consistency ruled in resistance fired metal using the 18mm radius tool; all test echelles except 3B and 3C used Silicone 704.

Test echelles 3A, 3B, 3C, were ruled in 2.5µm Au using Silicone 704, no oil, Cindol respectively to compare their relative merit in Au. Fig. 4 depicts these rulings which were made using a load of 29 grams suitable for Al but insufficient for Au. Fig. 4d shows a full depth ruling in the same coating; the load here was 40 grams. A third test echelle was ruled in this same coating with a 35 gram load. Efficiency data for Al replicas of these test echelles are reported in Appendix B as #4A1 and #3A1 respectively. The sharp diagonal line nearly centered in Fig. 4d is the groove bottom (diamond ruling edge) separating the smooth (bright) blaze face from the residually rough (dark) unblazed face of the groove. Please note that the unblazed face has been steepened and appears narrower than the blazed face in contrast to Fig. 4a.

Fig. 4 is interpreted to show:

- Groove depth was greatest with Cindol, intermediate with no oil, least with Silicone.
- Groove smoothness appeared better for Silicone than for Cindol or no lubricant; there did not seem to be a significant difference between dry ruling and Cindol.
- The full depth ruling picture shows as smooth a blaze face as any ever studied by Hyperfine. This test echelle was found to have the largest optical efficiency of any measured to this time.

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IVD. 10µm Coatings, 100 gr/mm Test Echelles

Fig. 5 contrasts 300 gr/mm grooves in 2.5 - 3µm metal (a, b, c) with 100 gr/mm grooves in 10µm metal (d, e, f). It also contrasts grooves ruled in Al (a, b, d, e) with grooves ruled in Au (c, f). The 300 gr/mm grooves were photographed at highest magnification.

Fig. 5 is interpreted to show:

- 1. The grooves in Au are very much smoother than the grooves in Al or in Al-Si alloy.
- The 100 gr/mm grooves in the Al-Si alloy (Fig. 5d) appear to be less rough than those in non-alloyed Al (Fig. 5e).
- 3. The lower frequency grooves in thicker (rougher) met exhibit more residual roughness in each case.
- 4. The blaze face of the grooves in the 10µm Au appear to be quite smooth, not as smooth as in the 2.5µm Au but nearly so.

V. Optical Efficiency

VA. Apparatus Description

A deuterium Durce was used with a Tropel monochromator Model N-2 to produce ultraviolet radiation with about 5A bandwidth for illuminating the test rulings. The test echelles were mounted in an Ebert configuration. The detector was a 1P28 RCA photomultiplier. Suitable apertures were used to prevent overfilling the mirror that focused the diffracted light on the detector. The electrometer was a Model 110 Laboratory Photometer manufactured by Pacific Photometric Instruments. This apparatus was used for the 300 gr/mm test echelles as reported herein. The bandwidth of this system was too large to permit the orders of the 100 gr/mm test echelles to be separated.

Fig. 13 is a photograph of the apparatus as used to measure the efficiency of the 100 gr/mm test echelles. A 4 watt Hg line source had to be substituted for the continuum deuterium source. The source is mounted at the left in Fig. 13; its radiation is processed by the monochromator shown as the elongated box supporting the source. Radiation leaving the monochromator is deflected by a plane mirror into the square concave mirror at the rear of the table. This mirror collimates the light and sends it onto the test echelle shown mounted on the rotary table in the foreground. The spectrum diffracted by the test echelle is contained by the circular concave mirror at the rear of the table and focused via a small plane mirror into the photomultiplier clamped on the table. The electrometer appears at the right.

The reference basis for establishing grating efficiency was a plane AI mirror. Therefore all efficiency values reported are relative to the specular reflectance of AI at the same wavelength. All test echelles measured had AI surfaces as the Au test echelles were measured only after replication in AI.

VB. Efficiency of 300 gr/mm Test Echelles

Fig. 14 shows the efficiency as a function of wavelength in the 24th order blaze (with the wings of the 23rd and 25th orders) for two aluminum masters #1 and #2 and their respective first-generation replicas (#1A1, #2A1). Master #1 was ruled in resistance fired aluminum using an 18mm radius diamond and Silicone 704 lubricant; its groove character appears in Fig. 3a. Master #2 was ruled in e-gun fired aluminum using an 18mm radius diamond and Silicone 704 lubricant; its groove character appears in Fig. 3e. The actual data are tabulated in Appendix B.

Fig. 15 shows the same interference orders of the first generation aluminum replica of the best test echelle ruled in the 2.5µm Au of Fig. 2b; grooves of this grating are shown in Fig. 4d. This test echelle was marked #4A1, and its data are given in Appendix B. This is a refinement of an earlier ruling (#3A1) in the same coating; data for this prior test echelle are also listed in Appendix B.

Fig. 15 shows that the 24th order of the test echelle whose grooves are shown in Fig. 4d had a maximum relative efficiency of 67% at .195µm, corresponding to 61% absolute efficiency. This is the highest efficiency Hyperfine has obtained, and the grooves were the smoothest yet. Neither the 23rd nor the 25th orders had as much as 5% efficiency at .195µm. The energy was predominantly concentrated in the 24th order.

VC. Efficiency of 100 gr/mm Test Echelles.

Data for the three 100 gr/mm test echelles were taken at four strong Hg lines between .2537 and .4047 um. The results are tabulated below:

Table II. Relative Efficiency Data for 100 gr/mm Test Echelle Replicas.

| 入 (μm) | Order | Aluminum (Fig 5e) | Al-Si (Fig. 5d) | Au (Alum. Replica) (Fig. 5f) |
|-----------|-------|----------------------|--------------------|---------------------------------|
| .2537 | 55 | 5 | 6 | 4 |
| | 56 | 39 | 35 | 21 |
| | 57 | 8 | 8 | 39 |
| | 58 | | | 5 |
| .3131 | 44 | | | 6 |
| | 45 | 30 | 30 | 17 |
| | 46 | 24 | 23 | 40 |
| | 47 | | | 6 |
| .3650 | 38 | 10 | 12 | 8 |
| | 39 | 34 | 34 | 42 |
| | 40 | 10 | 10 | 10 |
| .4047 | 34 | | 7 | |
| | 35 | 44 | 42 | |
| | 36 | 12 | 12 | 52 |
| | 37 | | 6 | |

The sum of the energy in the observed orders for the UV light is significantly larger for the AI replica of the gold test echelle than for the replicas of the AI test echelles, which we believe results from groove smoothness differences.

The data shows that the ultraviolet energy is not concentrated in a single order. This concentration is better for the Au grating than for the AI or AI-Si alloy test echelles, which may indicate that the £ u grooves are less curved.

The incomplete concentration of radiation in one spectrum order implies that the blaze surface of each groove is not accurately plane. For a given groove profile, the groove slope error increases with width. Moreover, the ruling of grooves 3 times deeper and wider in coatings that are less smooth may seriously compound the dispersal of radiation into several orders.

The data furthermore seems to show even less favorable concentration at shorter wavelengths. The extrapolation of this behavior to Lyman Alpha is evidence that there is much room for improvement.

VI. Conclusions

Observations of coating and test echelle characteristics have been reported throughout this report. Hyperfine accepts most of these observations only as working hypothesis and prefers not to treat them as conclusions. We believe it significant that the same test echelle had the smoothest grooves and the highest spectral efficiency in support of the intuitive feeling that smooth grooves are needed for high grating efficiency. We further believe that gold shows important promise as a metal in which master echelles for ultraviolet use could be ruled. 300 gr/mm test echelles can, with maximum care, have absolute efficiency in the 60% neighborhood for .2µm radiation.

The 100 gr/mm test echelles made to date fail to concentrate UV radiation in a single order. Here again, however, Au was better than AI or the AI-Si alloy.

POLEGOE FRAME

APPENDIX A — Table of Test Echelles and Coatings, cross-referenced to Electron-Micrographs and Efficiency Figure

| Coating (#) Echelle (∝) | Metal | Thick µm | Coating Method | Coating | Fig. Numbers Grooves | Efficiency |
|----------------------------|--------------------|------------------|-------------------|---------------|-------------------------|------------|
| 1A | Al | 3 | Res. | 1b | 3a | 14a, c |
| 1A' | | | | | 3b | |
| 1B | | | | and | 3c | |
| 1B' | | | | 2e | 3d | . 4 |
| 1C | Al + Au | 3 + .5 | + Res. | - | - | - |
| 2A | Al | 3 | e-gun | 1c | 3e | 14b, d |
| 2A' | Carle Ta | | | | 3f | |
| 2B | | | | | 3g | |
| 2B' | | | | | 3h | |
| 2C | Al + Au | 3 + .5 | + Res. | - | - | |
| 3A | Au | 2.5 | Res. | 2b | 4a | |
| 3B | Gertage - 1 | , translated the | | | 4b | |
| 3C | | ed-e-constant | en de Medica | lanumin ket v | 4c | |
| 3D | | | | | 4d | 15 |
| 4A | Au | 6 | Res. | | - | |
| 5A | AI | 10 | Res. | 1e, 2f | 5e | Tab. II |
| 6A | AI, Si | 10 | Res. | 1d | 5d | Tab. II |
| 7A | Au | 10 | Res. | 2c | 5f | Tab. II |
| ilia de esta de | g 20 - 20 - 10 - 1 | | | | of or a specific | |

For more information on the data contained in this chart, see the following page.

| LROUY. | EMAME | 2 |
|--------|-------|---|
| | | |

| g. Numbers Grooves | Efficiency | Frequency gr/mm | Oil | Tool r (mm) | Comments |
|-----------------------|------------|--------------------|------|----------------|---|
| 3a | 14a, c | 300 | Sil | 18 | Dow Corning Silicone 704 |
| 3b | | 300 | Sil | 6 | Dow Corning Silicone 704 |
| 3c | | 300 | Cin | 18 | Cindol 3401 |
| 3d | | 300 | Cin | 6 | Cindol 3401 |
| - | - | - | - | - | Al + Au interaction blistered |
| 3e | 14b, d | 300 | Sil | 18 | |
| 3f | | 300 | Sil | 6 | |
| 3g | - | 300 | Cin | 18 | |
| 3h | | 300 | Cin | 6 | |
| - | - | - | - | | Al + Au interaction blistered |
| 4a | | 300 | Sil | 18 | Ruled part depth with load appropriate for Al coating |
| 4b | | 300 | None | 18 | Ruled part depth with load appropriate for Al coating |
| 4c | | 300 | Cin | 18 | Ruled part depth with load appropriate for Al coating |
| 4d | 15 | 300 | Sil | 18 | Full depth ruling |
| | - | 300 | Sil | 18 | Tape test OK. Ruling tore up. |
| 5e | Tab. II | 100 | Sil | 18 | |
| 5d | Tab. II | 100 | Sil | 18 | Initial alloy 2% Si - 98% Al |
| 5f | Tab. II | 100 | Sil | 18 | |
| | | | | 200 | |

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Appendix A2

Coatings identified in the preceding table by the numbers 1, 2, and 3 (each with letter suffices) were made by Evaporated Metal Films, Inc. of Ithaca, N.Y. Coatings numbered 4, 5, 6 and 7 were made at Hyperfine in single pump-downs.

All of the echelles were ruled by either of two diamond tools made by the J. Robert Moore Co. of Petersham, Mass. in accordance with the specification of Fig. 6. The tools were symmetrical and had a 90° dihedral angle or ruling edge. The side profile of the ruling edge was an 18mm radius for one of the tools, 6mm for the other. The tools were each oriented to burnish the plane smooth, specular, "blazed" face of the V-groove at a 45° angle from the plane of the grating blank.

Successive trials and tests were used during each ruling set-up to achieve optimum full-depth grooves. There is a trade-off of residual unruled surface on the one hand and irregular burnishing burr on the other. Final records of the tool loading masses were not kept for each ruling. Approximate masses for gold were 40 grams for 300 gr/mm test echelles and 250 grams for the 100 gr/mm test echelles. The corresponding approximate masses used when ruling aluminum coatings and the 10µm aluminum-silicone alloy coating were 29 grams and 105 grams. The rulings into e-gun evaporated aluminum required only 22 grams for full depth at 300 gr/mm.

Coating runs 1 and 2 each contained three blanks (A, B, and C). The A blanks were ruled using Dow Corning Silicone 704, and the B blanks were ruled using Cindol 3401 (E.F. Houghton & Co.). Separate areas of each of the A blanks and each of the B blanks were ruled using the two diamond tools; this is indicated in the table by unprimed and primed letters.

The C blanks from coating runs 1 and 2 were overcoated by Evaporated Metal Films, Inc. in a third (unnumbered) coating run with .5µm gold. These coatings could not be ruled because the aluminum and gold interacted and the surface erupted with blisters.

APPENDIX B — 300 gr/mm Efficiency Data.

Aluminum masters and aluminum replicas (A1 suffix). Readings under 5% are omitted.

| | | Alumi | Gold | Gold Masters | | |
|---------|----|-------|------|---------------------|------|------|
| λ/Order | #1 | #1A1 | #2 | #2A1 | #3A1 | #4A1 |
| .187 | | | | | | |
| 23 | 10 | 10 | | | 7 | |
| 24 | 29 | 37 | 24 | | | |
| 25 | 30 | 28 | 45 | 41 | 61 | 65 |
| .188 | | | | | | |
| 23 | 4 | | | | | |
| 24 | 39 | 36 | 28 | 30 | | |
| 25 | 15 | 18 | 38 | 35 | 60 | 67 |
| .189 | | | | | | |
| 23 | 5 | | | | | |
| 24 | 42 | 43 | 36 | 37 | 21 | |
| 25 | | 14 | 33 | 29 | 50 | 57 |
| .190 | | | | | | |
| 23 | 5 | 5 | | | 5 | |
| 24 | 49 | 46 | 43 | 43 | 30 | 27 |
| 25 | 8 | 8 | 25 | 21 | 47 | 51 |
| .191 | | | | | | |
| 23 | 7 | 7 | 7 | | | |
| 24 | 51 | 46 | 48 | 47 | 38 | 37 |
| 25 | 4 | | 18 | 15 | 41 | 44 |
| .192 | | | | | | |
| 23 | 8 | 9 | | 4 | | |
| 24 | 48 | 41 | 52 | 50 | 49 | 50 |
| 25 | 4 | | 9 | 7 | 31 | 32 |

APPENDIX B — Continued

| | | Aluminun | Gold Masters | | | |
|---------|----|----------|---------------------|------|------|------|
| λ/Order | #1 | #1A1 | #2 | #2A1 | #3A1 | #4A1 |
| .193 | | | | | | |
| 23 | 13 | 14 | 12 | 8 | | |
| 24 | 45 | 47 | 54 | 50 | 58 | 60 |
| 25 | | | | | 20 | 20 |
| .194 | | | | | | |
| 22 | 8 | 7 | | | | |
| 23 | 19 | 25 | 15 | 15 | | |
| 24 | 38 | 38 | 51 | 49 | 60 | 64 |
| .195 | | | | | | |
| 23 | 26 | 35 | 20 | 21 | | |
| 24 | 30 | 30 | 47 | 48 | 61 | 67 |
| .196 | | | | | | |
| 23 | 38 | 37 | 27 | 29 | | |
| 24 | 15 | 18 | 40 | 37 | 60 | 64 |
| .197 | | | | | | |
| 23 | 41 | 38 | 33 | 35 | 19 | 15 |
| 24 | 15 | 14 | 33 | 29 | 55 | 60 |
| .198 | | | | | | |
| 23 | 46 | 50 | 37 | 41 | 27 | 20 |
| 24 | 8 | 10 | 25 | 23 | 48 | 53 |
| .199 | | | | | | |
| 23 | 50 | 54 | 45 | 45 | 33 | 34 |
| 24 | | 20 | | 17 | 40 | 45 |
| .200 | | | | | | |
| 23 | 51 | 52 | 50 | 50 | 42 | 42 |
| 24 | | | 14 | 12 | 34 | 34 |
| .225 | | | | | | |
| 20 | 30 | 36 | 23 | 28 | 12 | |
| 21 | 23 | 22 | 38 | 37 | 55 | 60 |
| .250 | | | | | | |
| 18 | 31 | 29 | 26 | 30 | 13 | 10 |
| 19 | 22 | 18 | 34 | 34 | 50 | 55 |
| | | | | | | |

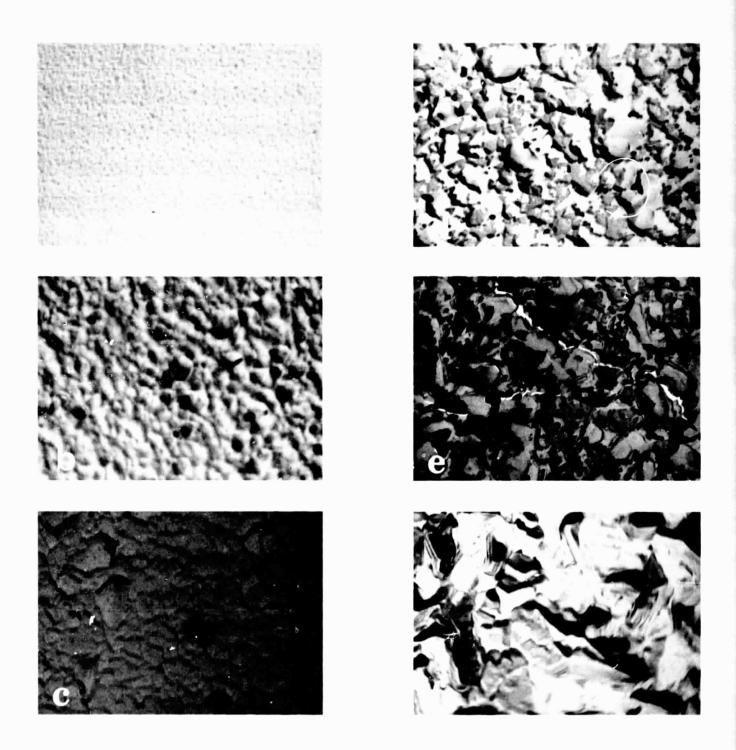


Figure 1, Aluminum coatings

- a) .3 μ m resistance, b) 3 μ m resistance, c) 3 μ m E-gun,
- d) 10 µm AI-Si (2%?) resistance, e) 10 µm resistance,
- f) 10 μ m E-gun. Mag = 14,000 X.

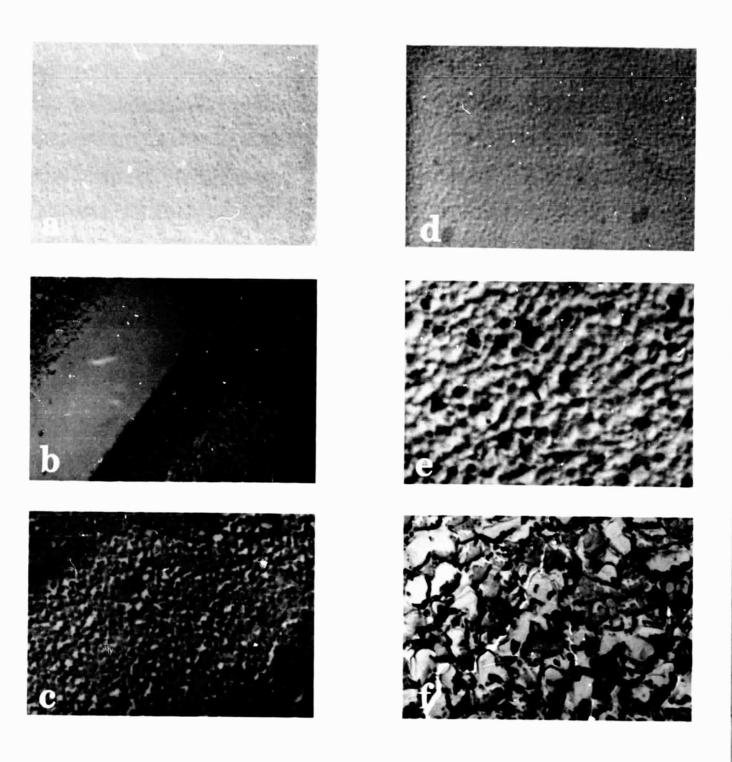


Figure 2, Au vs. Al resistance fired coatings a) .3 μ m Au, b) 2.5 μ m Au, c) 10 μ m Au, d) .3 μ m Al, e) 3 μ m Al, f) 10 μ m Al. Mag \cong 14,000 X

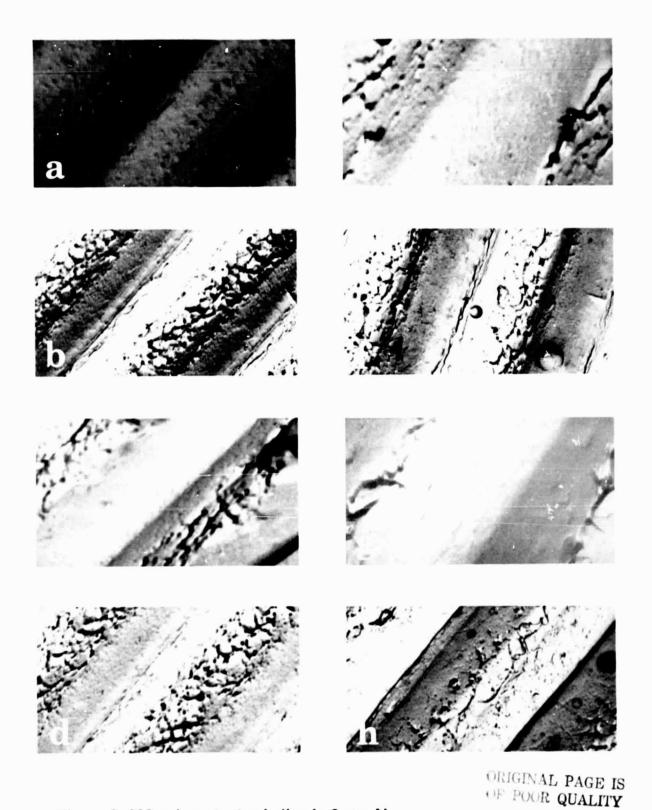


Figure 3, 300 gr/mm test echelles in 3 µm Al.

Resistance (a-d) and E-gun (e-h) ruled with Silicone 704 (a, b, e, f) and Cindol 3401 (c, d, g, h). The 18 mm radius tool was used for a, c, e, g; the 6 mm radius tool for b, d, f, h. Each groove is 3.33µm wide.

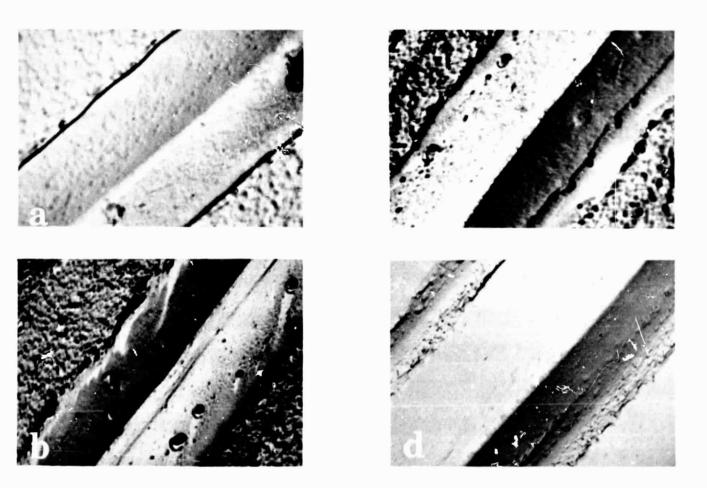


Figure 4, 300 gr/mm rulings in 2.5 μm Au.

- a) Silicone 704, b) no oil, c) Cindol 3401 ruled with underloaded diamond; note unruled surface. d) Full depth ruling with Silicone 704 best test echelle obtained.
- a, b and c ruled portion approximate $\frac{1}{2}$ depth = 1.666 μ m
- d, full depth 3.33µm wide.

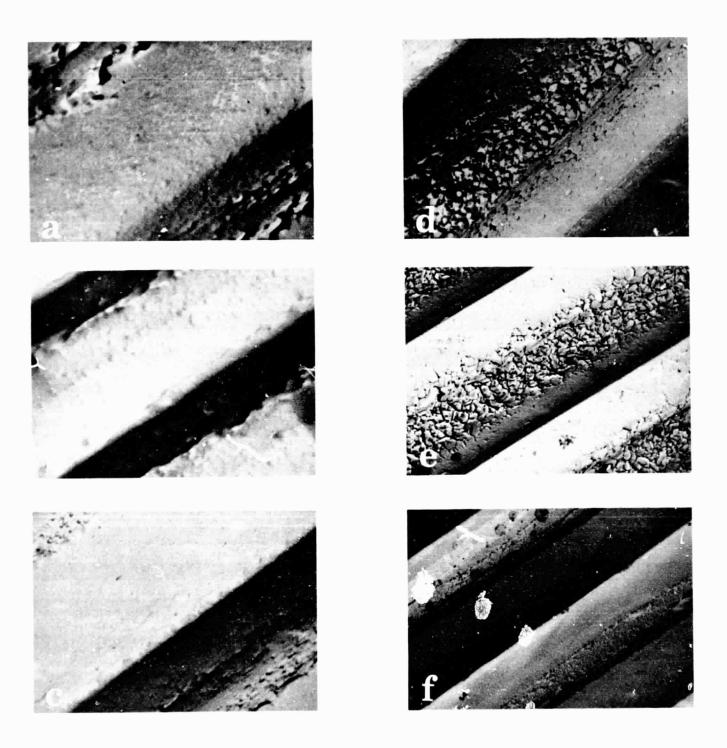


Figure 5, Al vs. Au rulings.

a) 300/mm in 3 µm E-gun AI, b) 300/mm in 3 µm resistance fired AI, c) 300/mm in 2.5 µm resistance fired Av, d) 100/mm in 10 µm resistance fired AI-Si (2%?) alloy, e) 100/mm in 10 µm resistance fired AI, f) 100/mm in 10 µm resistance fired Au. Groove width of a, b and c is 3.3µm; of d, e and f, 10µm.

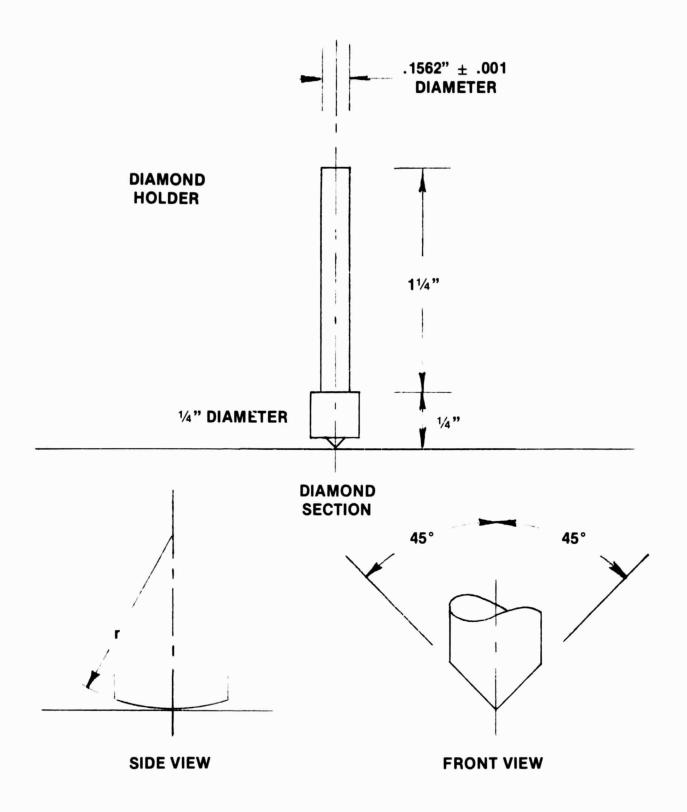


Figure 6, Diamond Tool Specification.

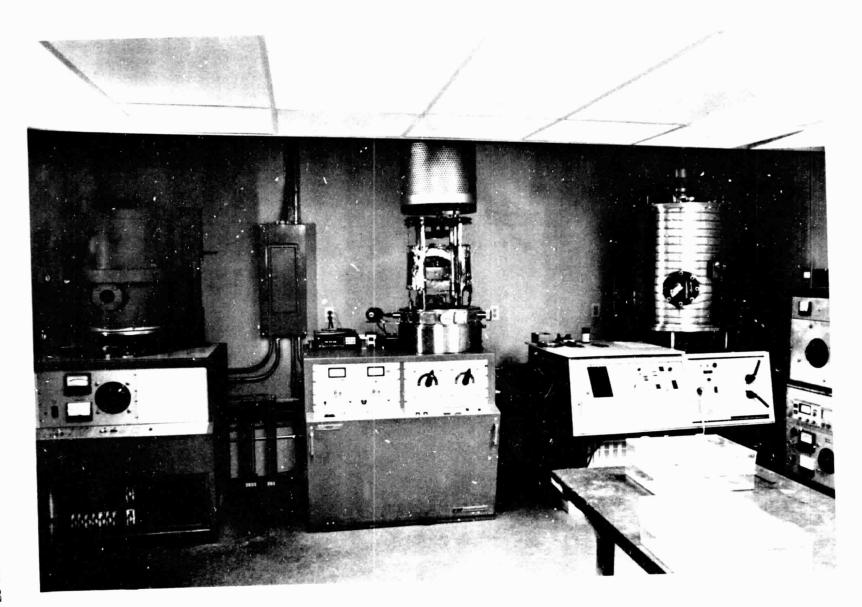


Figure 7, Vacuum system (open) used for coating master blanks.

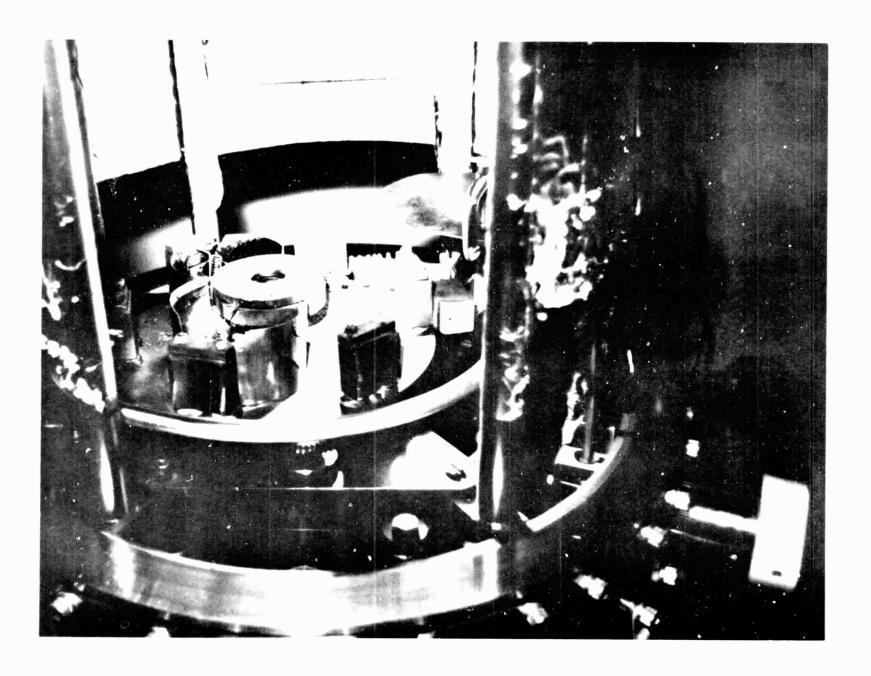


Figure 8, Turret filament assembly.



Figure 9, Planetary substrate holders, master blank, and monitor.

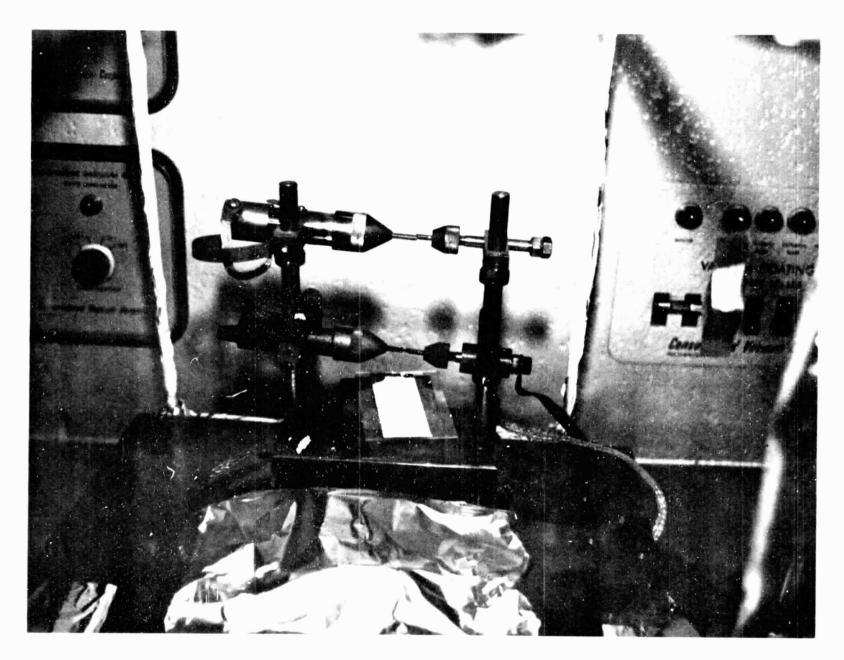


Figure 10, Coater used for electron micrograph replicas.

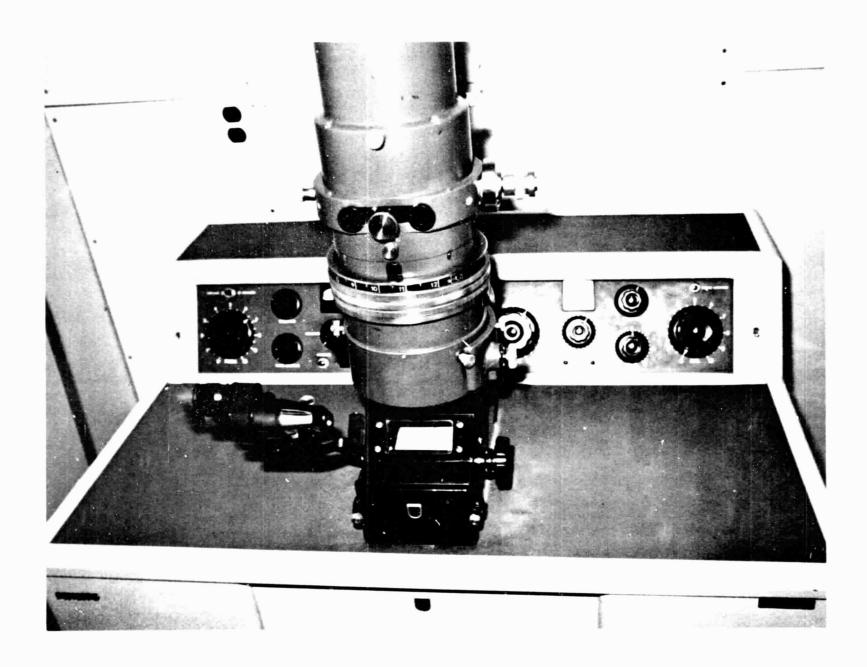


Figure 11, Electron microscope.

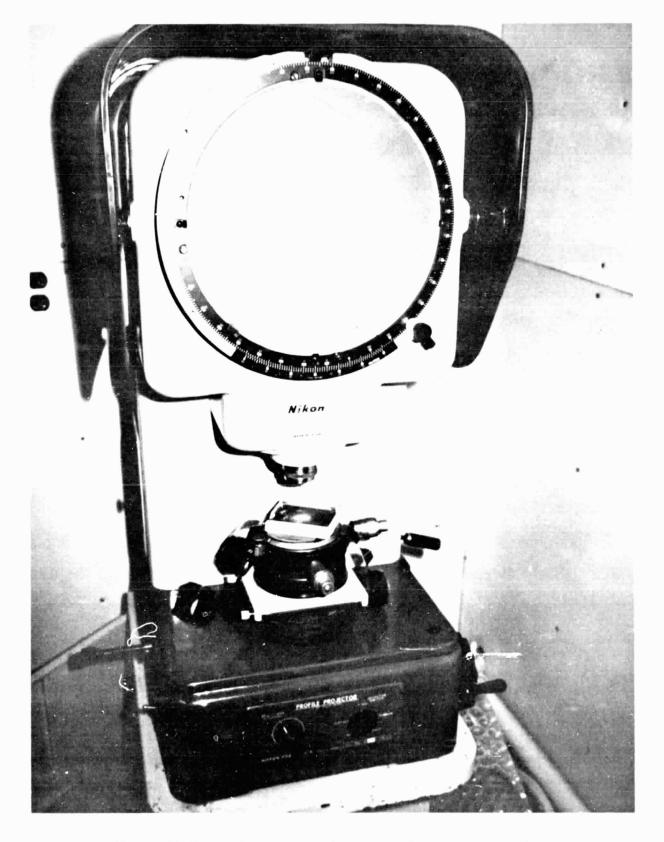


Figure 12, Projection system for measuring groove profiles.

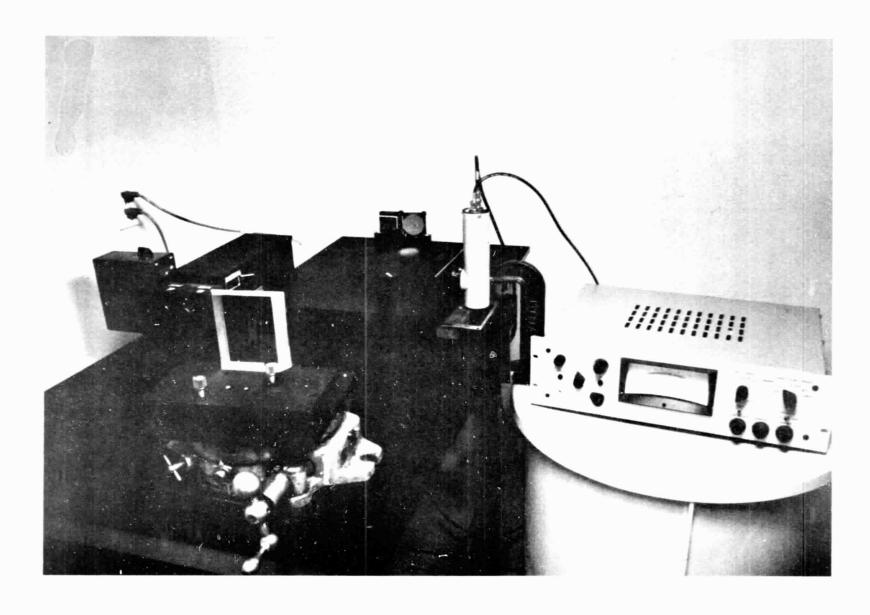


Figure 13, Efficiency measuring apparatus.

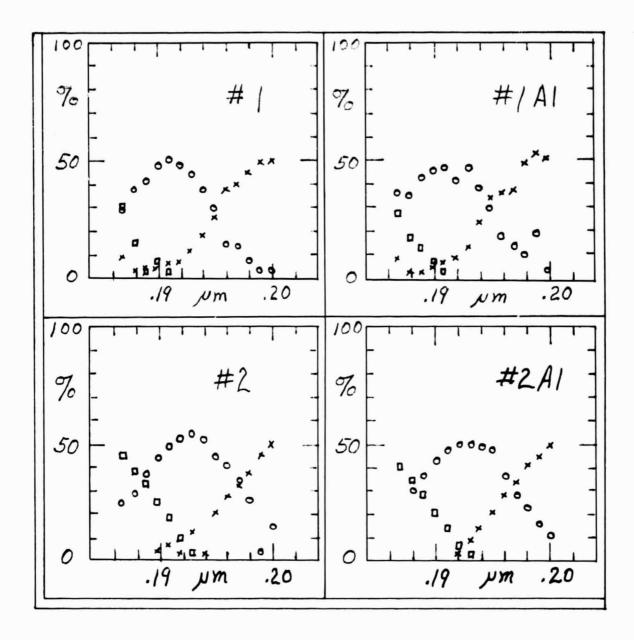


Figure 14, Relative efficiency of 23rd order (x), 24th order (o), and 25th order (\square) of 300 gr/mm aluminum test echelles and their first generation replicas.

#1 and #1A1 are for the master and replica of resistance fired aluminum. #2 and #2A1 are for the master and replica of e-gun fired aluminum.

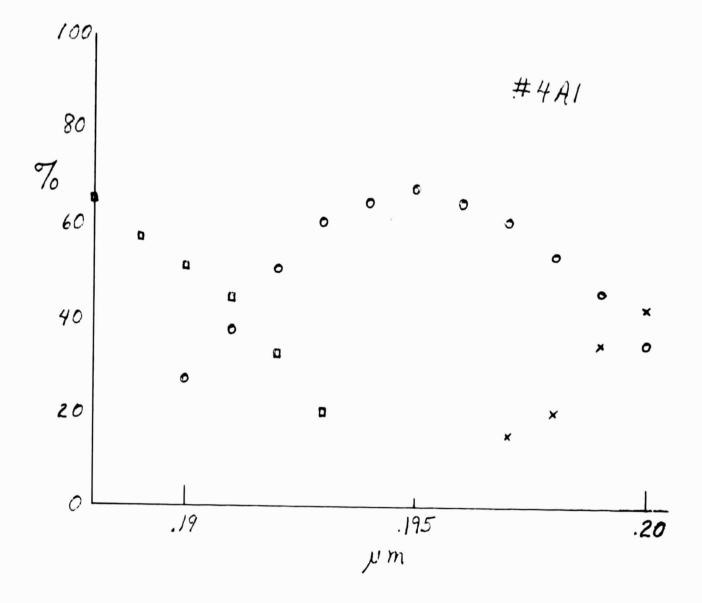


Figure 15, Relative efficiency of the first generation aluminum replica of the best test echelle ruled (Au master).

- x 23rd order
- o 24th order
- ☐ 25th order

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